PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
43050		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/US99/19458	26/08/1999	26/08/1998
International Patent Classification (IPC) or national classification and IPC C08K5/09		
Applicant THE DOW CHEMICAL COMPANY et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
01/03/2000	05. 12. 00
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Kaul-Buchberger, E Telephone No. +49 89 2399 8296

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/US99/19458

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*:
Description, pages:

1-19	as originally filed				

Claims, No.:

1-9	as received on	21/08/2000	with letter of	21/08/2000	

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   - ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
   - ☐ the language of publication of the international application (under Rule 48.3(b)).
   - ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   - ☐ contained in the international application in written form.
   - ☐ filed together with the international application in computer readable form.
   - ☐ furnished subsequently to this Authority in written form.
   - ☐ furnished subsequently to this Authority in computer readable form.
   - ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   - ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   - ☐ the description, pages:
   - ☐ the claims, Nos.:
   - ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-9
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-9
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-9
	No:	Claims	

2. Citations and explanations
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following documents:

   D1 = US-A-4254228
   D2 = US-A-5389696
   D3 = PATENT ABSTRACT OF JAPAN vol. 013, no. 479 (C-648) & JP-A-01188511

2.1. Document D1, which is considered to represent the most relevant state of the art, discloses (cf. column 2, lines 47 and 60-64, claim 1 and example 8) a process for the production moulded articles of polyurethane foam by forming a reaction mixture in a closed mould containing an organic polyisocyanate, a blowing agent, a polyol, a catalyst and a reaction product of an organic polyisocyanate with an ester of mixed ester of higher fatty acid which contains active hydrogen atoms as an internal mould release agent. The foaming mixture results in a moulded foam product which is removed from the mould.

2.2. The subject-matter of independent claims 1 and 6-8 differs from the disclosure of D1 in that an IMR-enhancer compound is present (see point VIII, items 1 and 2).

3.1. Comparison of examples 6-8 shows that the addition of a mineral oil as IMR-enhancer compound to the fatty acid condensation product leads to an improved release of polyurethane foam article, whereas the addition of mineral oil alone does not lead to the aforementioned effect.

3.2. The objective technical problem solved by the distinguishing feature is to improve the release of the polyurethane foam article.

3.3. Although D3 discloses that mineral oil release agents are suitable as internal mould release agents in the cast polymerization of a diisocyanate and a mercaptoalkyl ester, said document does not give a suggestion that the addition of mineral oil leads to the aforementioned effect.

3.4. Therefore, the subject-matter of independent claims 1 and 6-8 as well as of the dependent claims 2-5 and 9 fulfill the requirements of Article 33(3)PCT.

4. For all claims (1-9) industrial applicability is acknowledged.

Re Item VIII

Certain observations on the international application

1. Claim 6 does not define a method. Therefore, the wording "in accordance with the method of Claim **6**" in claim 8 is not correct and thus claim 8 is unclear and therefore said claim does not meet the requirements of Article 6 PCT.

2. In claims 1-5, 7 and 8 it is not clear which compounds are meant by the word "IMR-enhancer compound". In the national phase this word should be replaced by the corresponding concrete chemical compounds.

43050

Patent Application
Applicant: Ron H. Niswander
Express Mail No.: EL026307646US

What is claimed is:

1. A method for preparing a molded foam article comprising:
   a. forming a polyurethane-forming mixture by mixing
      i. a fatty acid condensation product;
      ii. an internal-mold-release-enhancer compound;
      iii. an isocyanate;
      iv. a polyol;
      v. a catalyst; and
      vi. a blowing agent;
   b. filling a mold with the polyurethane-forming mixture;
   c. forming a molded foam article; and
   d. removing the molded foam article from the mold.

2. The method of Claim 1, wherein the fatty acid condensation product, the internal-mold-release-enhancer compound, and the isocyanate are first mixed to yield an enhanced internal mold release "A" side composition.

3. The method of Claim 1, wherein the fatty acid condensation product, the internal-mold-release-enhancer compound, and the polyol are first mixed to yield an enhanced internal mold release "B" side composition.

4. The method of Claim 1, wherein a portion of the fatty acid condensation product, a portion of the internal-mold-release-enhancer compound, and the isocyanate are first mixed to yield an enhanced internal mold release "A" side composition and wherein the residual portion of the fatty acid condensation product, the residual portion of the

internal-mold-release-enhancer compound, and the polyol are mixed to yield an enhanced internal mold release "B" side composition.

5. The method of Claim 1, wherein the fatty acid condensation product is a condensation product of a fatty acid selected from the group consisting of ricinoleic acid, oleic acid, alaidic acid, stearic acid, palmitic acid, linoleic acid, octanoic acid, coconut oil acids, tallow fatty acid, paraffin oxidation acids, and tall oil fatty acid and the internal-mold-release-enhancer compound is mineral oil.

6. A molded foam article prepared in accordance with the method of Claim 1.

7. A method for preparing an enhanced internal mold release composition comprising:

a. reacting a fatty acid condensation product with an isocyanate in the presence of an internal-mold-release-enhancer compound,

wherein the fatty acid condensation product, having at least one active hydrogen containing group.

8. An enhanced internal mold release composition prepared in accordance with the method of Claim 6.

9. The method of Claim 1, wherein the components mixed in the "forming a polyurethane-forming mixture" step further comprise the enhanced internal mold release composition of Claim 7.

PATENT COOPERATION TREAT

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
43050		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/US 99/ 19458	26/08/1999	26/08/1998
Applicant		
THE DOW CHEMICAL COMPANY et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

   [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

   [ ] contained in the international application in written form.

   [ ] filed together with the international application in computer readable form.

   [ ] furnished subsequently to this Authority in written form.

   [ ] furnished subsequently to this Authority in computer readble form.

   [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

   [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

   [X] the text is approved as submitted by the applicant.

   [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

   [X] the text is approved as submitted by the applicant.

   [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. _____

   [ ] as suggested by the applicant.

   [ ] because the applicant failed to suggest a figure.

   [ ] because this figure better characterizes the invention.

   [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/19458

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08K5/09

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 013, no. 479 (C-648), 18 October 1989 (1989-10-18) & JP 01 188511 A (ASAHI OPTICAL CO LTD), 27 July 1989 (1989-07-27) abstract	1,5
A	US 4 965 293 A (SANNS JR. FRANK) 23 October 1990 (1990-10-23) column 7, line 16 -column 8, line 42; claims 1,12	1,3,5-7
A	US 4 868 224 A (S.J.HARASIN ET AL.) 19 September 1989 (1989-09-19) cited in the application column 2, line 58 -column 3, line 3; claim 1	1,6-9

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
9 December 1999	17/12/1999

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Angiolini, D

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INTERNATIONAL SEARCH REPORT

[redacted]ation on patent family members

[redacted]rnational Application No
[redacted]CT/US 99/19458

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 01188511 A	27-07-1989	NONE	
US 4965293 A	23-10-1990	CA 2017810 A	08-12-1990
US 4868224 A	19-09-1989	CA 1318451 A	25-05-1993
		EP 0364846 A	25-04-1990

INTERNAL MOLD RELEASE FOR LOW DENSITY REACTION INJECTION MOLDED POLYURETHANE FOAM

FIELD OF THE INVENTION

This invention relates to molded foams. More particularly, it relates to a method for preparing a molded polyurethane foam article using an internal mold release composition.

DESCRIPTION OF THE PRIOR ART

Molded polyurethane foams (hereinafter "molded foams") are useful in a variety of applications. For example, molded foams are useful in the construction of furniture, automobiles, and buildings. Molded foams are especially important because they are lightweight and resistant to moisture, weather, temperature extremes, and aging.

Molded foams are produced from foamable reaction mixtures. A foamable reaction mixture minimally contains of an organic polyisocyanate, an active hydrogen containing compound, a catalyst, and a blowing agent.

To obtain a molded foam article, a foamable reaction mixture is injected into a mold, wherein the mixture foams and then solidifies in a compacted state. The mixture takes on the mold's shape and features. Injection molding or reaction injection molding (RIM) processes can be used to prepare the molded foam article.

To facilitate removal of the molded foam article from the mold, an external mold

release (EMR) agent can be used to coat the mold. Examples of typically used EMR agents are waxes, soaps, and oils. The EMR agent reduces the mold opening force required to free the molded foam article from the mold.

Unfortunately, the use of EMR agents can have deleterious effects. Residue from the EMR agent can accumulate on the mold surface. The residue can obscure detailed features of the mold, thereby preventing proper imprinting of the features onto the surface of molded foam article. Also, the removal of the residue requires periodic cleaning of the mold. Periodic cleaning results in removing the mold from service, thereby affecting the service time of the mold.

Also, EMR agents can adhere to the surface of the molded foam article and is often removed from the mold surface with the molded foam article. The EMR agents must therefore be replaced to provide continued release of the molded foam articles. The necessity for repeated additions of EMR agent results in additional expense due to both time losses for applying additional quantities of EMR agent to the mold surfaces as well as the cost of the EMR agent itself.

The use of an internal mold release (IMR) agent can alleviate some of the problems associated with the use of an EMR agent alone. IMR agents can be included as integral components of a foamable reaction mixture. Examples of processes for preparing molded foams from a foamable reaction mixture, containing an IMR agent, are disclosed in U.S. Patent Nos. 4,201,847; 4,254,228; 4.868,224; 5,019,317; and 5,389,696. A foamable reaction mixture containing an organic polyisocyanate and an active hydrogen containing fatty acid ester is specifically disclosed in U.S. Patent Nos. 4,201,847 and 4,225,228. U.S. Patent No.

5,389,696 specifically discloses the use of an expensive mixture of fatty esters as an IMR agent.

Unfortunately, IMR agents do not generally function as well as EMR agents. Also, IMR agents can be expensive. Therefore, it would be desirable in the art of preparing molded foam articles to have an IMR composition that reduces the mold opening force required to free a molded foam article from a mold. It would also be desirable to have an IMR composition that reduces the amount of an EMR agent that needs to be applied to a mold's surface. Furthermore, it would be desirable to replace an IMR agent with a less expensive material without adversely affecting the opening force. Moreover, it would also be desirable to reduce the cycle time associated with the use of EMR agents.

SUMMARY OF THE INVENTION

According to the present invention, a method for preparing a molded polyurethane foam article using an internal mold release composition is provided. More specifically, a method using an IMR agent and an IMR-enhancer compound to prepare a molded polyurethane foam article is provided.

In its generic embodiment, the method comprises (a) forming a polyurethane-forming mixture by mixing (i) a fatty acid condensation product, (ii) an IMR-enhancer compound, (iii) an isocyanate, (iv) a polyol, (v) a catalyst, and (vi) a blowing agent; (b) filling a mold with the polyurethane-forming mixture; (c) forming a molded foam article; and (d) removing the molded foam article from the mold. The IMR-enhancer compound is any liquid petroleum product within the viscosity range of products called oils. A molded foam article prepared in accordance with the method is also provided.

In its preferred embodiment, the method comprises first mixing the fatty acid condensation product, the IMR-enhancer compound, and the polyol to yield an enhanced IMR "B" side composition, followed by admixing the other components and then performing steps b-d. In an alternate embodiment, the fatty acid condensation product and the IMR-enhancer compound can first be added to the isocyanate to yield an enhanced IMR "A" side composition.

In an alternate embodiment, a method for preparing an enhanced IMR composition comprising reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound, wherein the fatty acid condensation product has at least one active hydrogen containing group, is provided. An enhanced IMR composition prepared in accordance with the method of the alternate embodiment is provided.

DESCRIPTION OF THE INVENTION

In the generic embodiment of the present invention, the method comprises (a) forming a polyurethane-forming mixture by mixing (i) a fatty acid condensation product, (ii) an IMR-enhancer compound, (iii) an isocyanate, (iv) a polyol, (v) a catalyst, and (vi) a blowing agent; (b) filling a mold with the polyurethane-forming mixture; (c) forming a molded foam article; and (d) removing the molded foam article from the mold.

In the present invention, polyurethane refers a polyurethane compound obtained by the reaction of a polyol with an isocyanate.

Fatty acid condensation products suitable for the practice of the present invention can be prepared from fatty acids and alcohols, amino alcohols, amines or mixtures thereof. Useful fatty acids include saturated or unsaturated fatty acids. Preferred are aliphatic acids.

Suitable fatty acids include, but are not limited to, ricinoleic acid, oleic acid, alaidic acid, stearic acid, palmitic acid, linoleic acid, octanoic acids, coconut oil acids, tallow fatty acids, paraffin oxidation acids, and tall oil fatty acids. Preferred fatty acids are ricinoleic, oleic, adipic, and linoleic acids. The most preferred fatty acid is oleic acid. Diacids, such as adipic acid can also be used in the preparation of the fatty acid condensation product.

Several alcohols can be used to prepare esters suitable for practicing the present invention. Examples include butanol, hexanol, octanol isomers, dodecanol, oleyl alcohol, other fatty alcohols, natural or synthetic steroid alcohols, ethylene glycol, propylene glycol, butanediol, hexanediol, glycerol, polyglycerol, trimethylolpropane, pentaerythritol, sorbitol, hexitol, various sugars, and addition products of alkylene oxides such as ethylene oxide or propylene oxide. Preferred fatty esters can be obtained from the condensation of carboxylic acids and alcohols.

Several amines and amino alcohols are also useful in practicing the present invention. Examples include ammonia, monoalkylamines such as methylamine, dialkylamines such as diethylamine, and amine alkoxylation products such as ethanolamine.

Fatty acid esters of the present invention can be prepared by known methods. For example, a fatty acid ester suitable for the practice of the present invention can be prepared by condensation of an alcohol and a fatty acid at elevated temperatures. The temperature at which the reaction is conducted can be any temperature at which water is formed by reaction of the acid with the alcohol.

The reaction is preferably conducted at a temperature above about 100°C, more preferably above about 120°C, most preferably above about 150°C and less than about

200°C. The esterification reaction can optionally be conducted in a vacuum. Water can optionally be removed form the reaction mixture by azeotropic distillation.

A fatty acid condensation product is effective as IMR agent in amounts that allow removal of the molded foam article without destroying the article. An effective amount is in the range of from about 2 to about 15 wt %, based on the weight of the polyurethane-forming mixture. Preferably, the effective amount is within the range of from about 5 to about 10 wt%.

An IMR-enhancer compound of the present invention is any liquid product of petroleum within the viscosity range of products called oils. Examples of such oils are those commonly referred to as mineral oils. The preferred mineral oils are those known in the art as white mineral oils.

An IMR-enhancer compound is effective when it is added in amount sufficient to reduce the force to remove the molded foam article from the mold. The IMR-enhancer compound can be added in any amount within the range of from about 10 to about 100 wt % based on the weight of the fatty acid condensation product. Preferably, the IMR-enhancer compound is added in an amount within the range of from about 80 to about 90 wt%.

Any isocyanate or polyisocyanate known and used in the art of preparing molded foams can be suitable for the practice of the present invention, including aromatic and aliphatic isocyanates and polyisocyanates. The terms isocyanate and polyisocyanate are herein used interchangeably without limiting the scope of the individual terms. The isocyanate functionality can be present in an amount of from about 0.2 wt% to about 40 wt%.

U.S. Patent No. 4,785,027, incorporated herein by reference, discloses isocyanate compounds useful in practicing the present invention. Examples of useful isocyanates include the isomers of toluene diisocyanate (TDI) such as 2,4-toluene diisocyanate and 2,6-toluene diisocyanate, prepolymers of TDI, bis(4-isocyanatophenyl)methane (MDI), prepolymers of MDI, bis(isocyanatoethyl fumerate), dianisidine diisocyanate, toluidine diisocyanate, ethylene diisocyanate, 1,4-tetramethylene diisocyanate, 1,6-hexamethylene diisocyanate, 1,12-dodecane diisocyanate, cyclobutane-1,3-diisocyanate, 1,5-diisocyanato-3,3,5-trimethylcyclohexane, 2,4-hexahydrotoluene diisocyanate, 2,6-hexahydrotoluene diisocyanate, perhydro-2,4'-diphenylmethane diisocyanate, perhydro-2,6'-diphenylmethane diisocyanate, and mixtures thereof. Preferred isocyanate compounds include TDI, MDI, prepolymers of TDI, prepolymers of MDI, and mixtures thereof.

Any compound with multifunctional active hydrogen containing groups can be used in the practice of the present invention. The term "active hydrogen compound" refers to a compound that will react with an isocyanate to form an adduct between the active hydrogen containing compound and the isocyanate. Active hydrogen compounds can be described as compounds having functional groups that contain at least one hydrogen atom bonded directly to an electronegative atom such as nitrogen, oxygen or sulfur.

Polyols useful in practicing the present invention are generally known and are described in such publications as High Polymers, Vol. XVI, "Polyurethanes, Chemistry and Technology" by Saunders and Frisch, Interscience Publishers, New York, Vol. I, pp. 32-42, 44-54 (1962) and Vol. II, pp. 5-6,198-199 (1964); Organic Polymer Chemistry by K. J. Saunders, Chapman and Hall, London, pp. 323-325 (1973); Developments in Polyurethanes,

Vol. I, J. M. Burst, ed., Applied Science Publishers, pp. 1-76 (1978); and Reaction Polymers, W. Gum, W. Riese, and H. Ulrich, Eds., Hanser Publishers (1992), incorporated herein by reference. Suitable polyols have molecular weights of less than about 6000.

Examples of such polyols include (a) alkylene oxide adducts of polyhydroxyalkanes; (b) alkylene oxide adducts of non-reducing sugars and sugar derivatives; (c) alkylene oxide adducts of polyphenols; and (d) alkylene oxide adducts of amines and amine polyols.

Alcohols and amines are examples of other active hydrogen compounds that are useful in practicing the present invention.

Examples of catalysts useful in practicing the present invention include tertiary amine catalysts such as triethylene diamine, N-methyl morpholine, N-ethyl morpholine, diethylethanolamine, N-coco morpholine, 1-methyl-4-dimethylaminoethyl piperazine, and bis(N,N-dimethylaminoethyl)ether. Amine catalysts are usually used in an amount of from about 0.1 to about 5, preferably from about 0.2 to about 2 parts per 100 parts of polyol formulation.

Organometallic catalysts are also suitable. Examples include organolead, organoiron, organomercury, organobismuth, and preferably organotin compounds. Most preferred are organotin compounds such as dibutyltin dilaurate, dimethyltin dilaurate, stannous octoate, stannous chloride and similar compounds. Organometallic compounds are usually used in an amount from about 0.05 to about 2.0 parts per 100 parts of active hydrogen containing compound formulation.

Blowing agents useful for practicing the present invention include any gas or any material that generates a gas under the conditions of the reaction of the active hydrogen

containing compound with an isocyanate. Examples of blowing agents include carbon dioxide, water, low-boiling hydrocarbons, halogenated hydrocarbons, and nitrogen-releasing azo-compounds. Suitable low-boiling hydrocarbons include pentane, hexane, heptane, pentene, and heptene. Suitable halogenated hydrocarbons include dichlorodifluoromethane, trichlorofluoromethane, 1,1,1-trichloroethane, and methylene chloride. Water and mixtures with low boiling hydrocarbons or halogenated hydrocarbons are preferred.

The "B" side of the present invention can include other components, such as copolymer polyols, surfactants, mold release agents (not of the present invention), fillers, flame retardants, and reinforcing fibers.

Examples of copolymer polyols suitable for practicing the present invention are disclosed in U.S. Patent No. 4,581,418, incorporated herein by reference. Suitable copolymer polyols are derived from polymerizable ethylenically unsaturated monomers. Examples of monomers used to prepare copolymer polyols useful in the present invention include aliphatic conjugated dienes such as butadiene; monovinylidine aromatics such as styrene, α-methylstyrene and vinylnapthalene, including other inertly substituted styrenes; α, β-ethylenically unsaturated carboxylic acids and esters such as acrylic acid, methacrylic acid, 2-hydroxyethacrylic acid and other similar compounds; α, β-ethylenically unsaturated nitriles such as acrylonitrile; acrylamide; vinyl esters such as vinyl acetate; vinyl ethers; vinyl ketones; and vinyl and vinylidene halides.

Examples of surfactants include silicone surfactants, polyethylene glycol ethers of long chain alcohols, tertiary amine or alkanolamine salts of long chain alkyl sulfate esters, alkyl sulfonic esters and alkylaryl sulfonic acids. Silicone surfactants include block

copolymers containing at least one polyoxyalkylene segment and one poly(dimethylsiloxane) segment.

U.S. Patent Nos. 4,868,224; 5,019,317; and 5,389,696, incorporated herein by reference, disclose examples of IMR agents which can be included in the "B" side of the present invention.

The present invention can also use crosslinkers. Examples of crosslinkers include diethanolamine and methylene bis(o-chloroaniline).

In its preferred embodiment, the method comprises first mixing the fatty acid condensation product, the IMR-enhancer compound, and the polyol to yield an enhanced IMR "B" side composition, followed by admixing the other components and then performing steps b-d of the generic embodiment. In an alternate embodiment, the fatty acid condensation product and the IMR-enhancer compound can first be added to the isocyanate to yield an enhanced IMR "A" side composition. A molded foam article is prepared in accordance with the methods of the present invention.

In another alternate embodiment, a portion of the fatty acid condensation product, a portion of the IMR-enhancer compound, and the isocyanate are first mixed to yield an enhanced IMR "A" side composition and the residual portion of the fatty acid condensation product, the residual portion of the IMR-enhancer compound, and the polyol are mixed to yield an enhanced IMR "B" side composition. Then, steps b-d are performed.

In an alternate embodiment, a method for preparing an enhanced IMR composition comprises reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound, wherein the fatty acid condensation product has at least one active

hydrogen containing group. In this embodiment, an enhanced IMR composition is prepared in accordance with the method of the alternate embodiment. Moreover, the enhanced IMR composition is admixed during the "forming a polyurethane-forming mixture" of the generic embodiment of the present invention.

The embodiments described herein are given to illustrate the scope and spirit of the present invention. The embodiments herein will make apparent to those skilled in the art other embodiments that may also be used. These other embodiments are within the scope of the present invention. Thus, the scope of the invention should be determined by the appended claims and their legal equivalents, rather than by the embodiments given herein.

EXAMPLES

The following examples merely exemplify various embodiments of the invention. It is understood that the following examples are provided to further illustrate the invention. They do not in anyway limit the scope of the present invention.

Table of Compounds

The following compounds were used in the preparation of the exemplified dispersions. The table provides the compound's generic name, its trade mark name, and its vendor.

Generic Name	Trademark	Vendor
propylene oxide polyol, 165 molecular weight	VORANOL™ 2025 Polyol	The Dow Chemical Company
propylene oxide polyol, 1000 molecular weight	VORANOL™ 230-112 Polyol	The Dow Chemical Company
propylene oxide polyol, 165 molecular weight	VORANOL™ 230-660 Polyol	The Dow Chemical Company

Generic Name	Trademark	Vendor
propylene oxide polyol initiated with ethylene diamine, 340 molecular weight	VORANOL™ RA640 Polyol	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bisphenyl isocyanate	PAPI™ 27 MDI	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bis-phenyl isocyanate	PAPI™ 94 MDI	The Dow Chemical Company
polymethylene polyphenyl isocyanate containing 4,4'-methylene bis-phenyl isocyanate	PAPI™ 95 MDI	The Dow Chemical Company
33% triethylene diamine in dipropylene glycol	Dabco 33LV	Air Products and Chemicals Inc.
methyl azanorbornane	Dabco AN10	Air Products and Chemicals Inc.
pentamethyl diethylene triamine	Polycat 5	Air Products and Chemicals Inc.
polyether-modified polysiloxane	Tagostab B8418	The Goldschmidt Company
polyether-modified polysiloxane	Tagostab 8427	The Goldschmidt Company
Complex ester of pentaerythritol, adipic acid, and oleic acid with low hydroxyl content	Loxiol G71S	The Henkel Corporation
polyethylene wax	Chem Tren 2004	Chem Tren
decaglycerol tertaoleate	Drewpol 10-4-O	Stepan
decaglycerol decaoleate	Polyaldol DGDO	Lonza Speciality Chemicals
tallamidopropyl dimethylamine	Shercodine T	Scher Chemicals
tallamidopropyl dimethylamine	Lexamine T-13	Inolex Chemical Company

Generic Name	Trademark	Vendor
tallaic salt of tallamidopropyl dimethylamine	Lexamine T-13T	Inolex Chemical Company
pentaerythritol oleate adipate	Inolex POA	Inolex Chemical Company

EXAMPLE NOS. 1-10

For Example Nos. 1 - 10, molded foam articles were prepared. The molded foam articles were prepared with "A" and "B" side formulations. The IMR-enhancer compound was added to the "B" side formulations, in accordance with the preferred embodiment of the present invention.

A heated mold was used to form the molded form article. The mold cavity was made of aluminum and maintained at 120°F (49°C). The cavity was approximately 3" (7.62 cm) in diameter with a 1" (2.54cm) x 3° draw. The mold core was made of stainless steel and maintained at 150°F (66°C).

A single light coating of external mold release (Chem Tren 2004 polyethylene wax) was applied to the core. When closed, the mold had a gap of 0.17" (cm). With the mold open, a 7"x7"x0.75" (17.8 cm x 17.8 cm x 1.9 cm) polyvinylchloride (PVC) coverstock sheet was placed over the cavity, and a one ounce (28.4 gm) glass mat was placed on top of the PVC.

The polyol formulation ("B" side) was mixed with the polymeric isocyanate ("A" side) in a desired ratio and poured onto the PVC sheet overlaid with glass mat. The mold was closed for 2 minutes, then opened and the molded foam article was removed by simply

pulling it off of the core.

The force required to remove the molded foam article from the mold was measured qualitatively. A value of 1 indicated that no force was required, 2 indicated that some force was required, 3 indicated that a lot of force required, 4 indicated that some type of tool required to loosen the molded foam article, and 5 indicated that the molded foam article strongly adhered to the mold and that the article could not be removed without damage to the molded foam article.

Successive quantities of the polyurethane-forming mixture were added to the mold until (1) a value of 4 was achieved or (2) at least twelve molded foam articles were prepared. Each set of quantities was formed into molded foam articles, which together make up an Example. The external mold release agent was not replenished between the successive additions. The release value was recorded through preparation of the twelfth article.

1. Example Nos. 1 and 2

Example No. 1 was prepared using 45% PAPI™ 94 MDI, 45% PAPI™ 95 MDI, and 10% Polyaldol DGDO on the "A" side. The "B" side formulation contained 60 parts VORANOL™ 2025 polyol, 30 parts VORANOL™ 230-112 polyol, 5 parts glycerine, 1.5 parts water, 2.0 parts Dabco 33LV, 0.5 parts Tagostab 8427, and 1.0 parts Polycat 5. The ratio of A:B was 1.8:1.

Example No. 2 was prepared using the same formulations as in Example No. 1, except (1) 2.0 parts Tagostab B8418 replaced the 0.5 parts Tagostab 8427 and (2) 14 parts mineral oil was added on the "B" side. The ratio of A:B was 1.5:1.

Articles for Example No. 1

Article No.	Release Value
1	1
2	1
3	1.5
4	2
5	3
6	4

Articles for Example No. 2

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1.5
6	1.5
7	2
8	3
9	4

A comparison of Example Nos. 1 and 2 clearly shows the advantage of using mineral oil as an IMR-enhancer compound.

2. Example Nos. 3 and 4

Example No. 3 was prepared using 100% PAPI™ 27 MDI on the "A" side. The "B" side formulation contained 42 parts VORANOL™ 2025 polyol, 33.6 parts VORANOL™ 230-112 polyol, 8.4 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.82 parts oleic acid, 3.66 parts Lexamine T-13, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, 12.0 parts mineral oil, and 14.0 parts Loxiol G71S. The ratio of A:B was 1.2:1.

Example No. 4 was prepared using 100% PAPI™ 94 MDI on the "A" side. The "B" side formulation was the same as used in Example No. 3, except (1) tall oil acid replaced

oleic acid, (2) Shercodine T replaced Lexamine T-13, and (3) mineral oil was not added. The ratio of A:B was 1.35:1.

Articles for Example No. 3

Article No.	Release Value
1	1.5
2	1.5
3	1.5
4	1.5
5	1.5
6	1.5
7	1.5
8	1
9	1
10	1.5
11	1
12	1.5

Articles for Example No. 4

Article No.	Release Value
1	1
2	1
3	1.5
4	1.5
5	1.5
6	2
7	2
8	2
9	2
10	2
11	2
12	2

The data shows that the absence of mineral oil makes removal of the article from the mold more difficult.

3. Example No. 5

Example No. 5 was prepared using 100% PAPI™ 27 MDI on the "A" side. The "B" side formulation contained 42 parts VORANOL™ 2025 polyol, 33.6 parts VORANOL™

230-112 polyol, 8.4 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.82 parts tall oil acid, 3.66 parts Shercodine T, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, and 12.0 parts mineral oil. The ratio of A:B was 1.35:1.

Articles for Example No. 5

Article No.	Release Value
1	1
2	1.5
3	2
4	4

The data indicates that mineral oil alone is not an effective internal mold release agent.

4. Example Nos. 6-8

Example No. 6 was prepared using 100% PAPI™ 94 MDI on the "A" side. The "B" side formulation contained 50 parts VORANOL™ 230-660 polyol, 30 parts VORANOL™ 230-112 polyol, 8.0 parts glycerine, 2.0 parts water, 1.5 parts Dabco 33LV, 2.0 parts Tagostab B8418, 1.5 parts Polycat 5, and 30 parts Loxiol G71S. The ratio of A:B was 1.3:1.

Example No. 7 was prepared using the formulations of Example No. 6, except the "B" side formulation also contained 14 parts mineral oil. The ratio of A:B was 1.15:1.

Example No. 8 was prepared using the formulations of Example No. 6, except (1) the Loxiol G71S was not added and (2) 14 parts mineral oil was added. The ratio of A:B was 1.45:1.

Articles for Example No. 6

Article No.	Release Value
1	1.5
2	1.5
3	1.5
4	1.5
5	1.5
6	1.5
7	1.5
8	1.5
9	1.5
10	1.5
11	1.5
12	1.5

Articles for Example No. 7

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1
6	1
7	1
8	1
9	1
10	1
11	1
12	1

Articles for Example No. 8

Article No.	Release Value
1	1
2	1.5
3	2
4	4

This data shows the advantage of using a mineral oil as an IMR-enhancer compound.

5. Example Nos. 9 and 10

Example No. 9 was prepared using 100% PAPI™ 94 MDI on the "A" side. The "B" side formulation contained 50 parts VORANOL™ 2025 polyol, 40 parts VORANOL™ 230-112 polyol, 10 parts glycerine, 2.0 parts water, 8.34 parts Lexamine T-13T, 0.26 parts oleic acid, 2.0 parts Tagostab B8418, 1.5 parts Dabco AN10, and 30 parts Inolex POA. . The ratio of A:B was 1.2:1.

Example No. 10 was prepared using the formulations of Example No. 9, except (1) tall oil acid replaced oleic acid and (2) 14.0 parts mineral oil was added to the "B" side formulation. The ratio of A:B was 1.6:1.5.

Articles for Example No. 9

Article No.	Release Value
1	1
2	1
3	1
4	1
5	1.5
6	4
7	2
8	4
9	4

Articles for Example No. 10

Example No.	Release Value
1	1
2	1
3	1
4	1
5	1
6	1
7	1.5
8	2
9	2
10	3
11	4
12	4

This data shows the advantage of using an IMR-enhancer compound such as mineral oil.

What is claimed is:

1. A method for preparing a molded foam article comprising:
   a. forming a polyurethane-forming mixture by mixing
      i. a fatty acid condensation product;
      ii. an IMR-enhancer compound;
      iii. an isocyanate;
      iv. a polyol;
      v. a catalyst; and
      vi. a blowing agent;
   b. filling a mold with the polyurethane-forming mixture;
   c. forming a molded foam article; and
   d. removing the molded foam article from the mold.

2. The method of Claim 1, wherein the fatty acid condensation product, the IMR-enhancer compound, and the isocyanate are first mixed to yield an enhanced IMR "A" side composition.

3. The method of Claim 1, wherein the fatty acid condensation product, the IMR-enhancer compound, and the polyol are first mixed to yield an enhanced IMR "B" side composition.

4. The method of Claim 1, wherein a portion of the fatty acid condensation product, a portion of the IMR-enhancer compound, and the isocyanate are first mixed to yield an enhanced IMR "A" side composition and wherein the residual portion of the fatty acid condensation product, the residual portion of the IMR-enhancer compound, and the

polyol are mixed to yield an enhanced IMR "B" side composition.

5. The method of Claim 1, wherein the fatty acid condensation product is a condensation product of a fatty acid selected from the group consisting of ricinoleic acid, oleic acid, alaidic acid, stearic acid, palmitic acid, linoleic acid, octanoic acid, coconut oil acids, tallow fatty acid, paraffin oxidation acids, and tall oil fatty acid and the IMR-enhancer compound is mineral oil.

6. A molded foam article prepared in accordance with the method of Claim 1.

7. A method for preparing an enhanced IMR composition comprising:

a. reacting a fatty acid condensation product with an isocyanate in the presence of an IMR-enhancer compound,

wherein the fatty acid condensation product, having at least one active hydrogen containing group.

8. An enhanced IMR composition prepared in accordance with the method of Claim 6.

9. The method of Claim 1, wherein the components mixed in the "forming a polyurethane-forming mixture" step further comprise the enhanced IMR composition of Claim 7.

INTERNATIONAL SEARCH REPORT

Inte ional Application No
PCT/US 99/19458

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08K5/09

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 013, no. 479 (C-648), 18 October 1989 (1989-10-18) & JP 01 188511 A (ASAHI OPTICAL CO LTD), 27 July 1989 (1989-07-27) abstract	1,5
A	US 4 965 293 A (SANNS JR. FRANK) 23 October 1990 (1990-10-23) column 7, line 16 -column 8, line 42; claims 1,12	1,3,5-7
A	US 4 868 224 A (S.J.HARASIN ET AL.) 19 September 1989 (1989-09-19) cited in the application column 2, line 58 -column 3, line 3; claim 1	1,6-9

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
9 December 1999	17/12/1999

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 99/19458

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